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Mass emission rate determinations were made by EPA reference methods. Cascade impactors were used to collect in-stack samples for particle size distribution measurements. Particulate samples for chemical characterization were collected on membrane filters for analysis by X-ray fluorescence spectroscopy. Development of measurement techniques required to determined the elemental composition of the total mass and sized fractions of the emission are discussed. Results of the tests at the three smelters include total mass and elemental emission rates, particle size distribution, and the elemental composition of the total particulate mass and of sized fractions from both the smelter furnaces and acid plants.

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Characterization of Particulate Emissions from Non-ferrous Smelters

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Chemical composition and particle size data for particulate emissions from stationary sources are required for environmental health effect assessments, air chemistry studies and for air quality modeling investigations such as source apportionment. The information presented in this paper is directed to those individuals concerned with these environmental investigations. In this study, particulate emissions from a group of non-ferrous smelters have been physically and chemically characterized. Emission samples were collected at the baghouse outlets from smelter furnaces and at smelter acid plant stacks at three locations; a zinc, a lead, and a copper smelter.

Mass emission rate determinations were made by EPA reference methods. Cascade impactors were used to collect in-stack samples for particle size distribution measurements. Particulate samples for chemical characterization were collected on membrane filters for analysis by X-ray fluorescence spectroscopy. Development of measurement techniques required to determine the elemental composition of the total mass and sized fractions of the emission are discussed. Results of the tests at the three smelters include total mass and elemental emission rates, particle size distribution, and the elemental composition of the total particulate mass and of sized fractions from both the smelter furnaces and acid plants. The results obtained at the copper smelter may not be representative of the emissions at the many copper smelters where reverbatory furnaces have been replaced.

Physical and chemical characterization of particulate emission from stationary sources are needed to provide information for many environmental studies including health effects, source apportionment and air chemistry. A characterization study has been conducted on three non-ferrous operations: a zinc, a lead, and a copper primary smelter. Particulate emission measurements were made on each smelter at stacks of two facilities; the furnace and the acid plant.

These studies were made as part of a program to develop measurement and characterization methods for emissions from stationary sources. Development of source sampling and analytical procedures are required to support emissions standards that are promulgated, to support air quality modeling studies such as dispersion modeling and source apportionment, to assess environmental impact, and to evaluate efficiency of control devices. Previous sources which have been investigated in this program include oil-fired power plants,[1] coal-fired power plants,[2] cement plants[3] and municipal sludge incinerators.[4]

Sampling Locations

Zinc Smelter

The first smelter where emissions were tested processes zinc ore and concentrates into metallic zinc and various zinc products, including high grade zinc oxide. Particulate emission testing was conducted at the stack of a vertical retort furnace and at the outlet side of a baghouse controlling emissions from a sintering machine at the acid plant. Combustion products from the retort furnace are exhausted through an air preheater and then to the atmosphere through a 150 foot stack. Sampling was conducted through two ports located in the circular stack.

At the acid sinter plant, the fumes driven off during the sintering process pass through a set of twin cyclones and a baghouse. The effluent from the baghouse was sampled through ports in the breeching between the baghouse and a 360 foot vertical stack.

Lead Smelter

The lead smelter that was tested converts lead concentrates to marketable pig lead. The first main process is the sinter plant where sulfur is removed from the concentrate and where the sinter suitable for blast furnace operation is formed. The sulfur dioxide that is formed is sent to the acid plant for conversion to sulfuric acid. Sinter and coke are fed into the top of the blast furnace where the sinter is converted to molten lead.

Testing was conducted at an outlet of the baghouse which controls emissions from the blast furnace and at the combined outlet duct of the acid plant and two baghouses which control emissions from the sinter plant.

Copper Smelter

At the copper smelter, concentrate is fed into reverbatory furnaces where it is smelted at approximately 2700°F. The matte, containing copper, iron and sulfur, is transferred to the converters. Silica flux is added to the converter bath where it combines with iron oxides to form slag which is returned to the reverbatory furnaces. Compressed air oxidizes the sulfur into sulfur dioxide and leaves metallic or blister copper. The emission gases from the converters are passed to the sulfuric acid plant.

Total mass emissions, characterization, and particle sizing samples were collected at two locations: the reverbatory furnace stack and the acid plant stack.

Sample Collection and Analysis

Sampling Procedures

The particulate sampling conducted at the primary smelters consisted of total particulate emissions (EPA Method 5), characterization samples on filters for chemical composition determination, and particle size determination by inertial impactors. Preliminary tra-

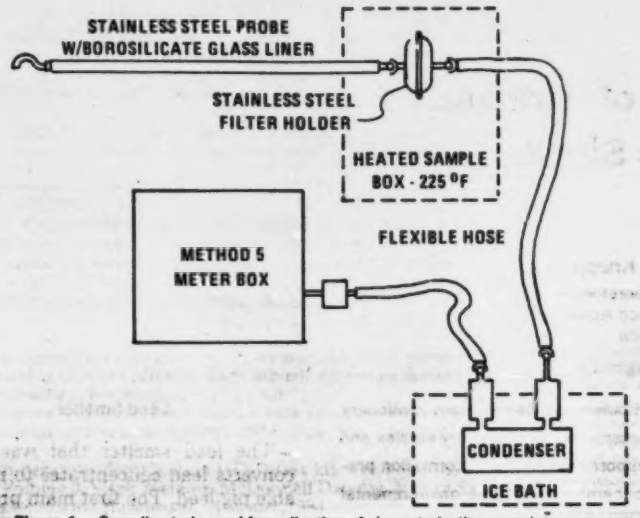

Figure 1. Sampling train used for collection of characterization samples.

verse measurements of the stack velocities were made to determine the velocity profiles and the required nozzle sizes for the impactors in order to provide isokinetic sampling. All tests were conducted at a single point of average velocity in the stack. Sampling for chemical composition was made at a single point because the X-ray fluorescence analyses require lightly loaded filters, thus the short sampling times made traversing during these sample collections impractical. Particle size sampling was also made at the same point because it is necessary to have a constant flow through the impactors in order to maintain the size cut-points. The total particulate sampling was made at the same point as the characterization and size samplings so that all would more closely represent the same emissions.

Total particulate emissions. Standard EPA Method 5 sampling trains[5] were used to determine stack particulate concentration at each of the three smelters tested. The Method 5 reference procedure was used, except sampling was conducted at a single point within the stack of the average velocity that was determined by the preliminary velocity traverse.

Characterization samples. Samples used for chemical characterization of the smelter emissions by X-ray fluorescence analysis (XRF) were collected with a modified Method 5 sampling train in which the conventional sample box, filter holder, and glass impingers are replaced by an EPA designed heated sample box that housed a stainless steel 47 mm filter holder (Figure 1). Collection media used for the characterization tests included Millipore AA, glass fiber, Nuclepore 0.8 μm, and fluorocarbon 0.2 μm filters. Sampling periods varied from 30 s to 5 min to provide a range of emission loadings on the filters. Several filters of each type were used to sample the stack stream but fluorocarbon filters were found to provide the best substrate for XRF analysis due to their chemical inertness, lack of particle penetration deep into filter, and adequate loading capacity without increase in pressure drop. In order to collect sulfuric acid aerosol to the same degree as Method 5, the filters were maintained at the same temperature, 225°F ± 20°F, in the heated sample box. The characterization tests were run simultaneously with the Method 5 tests.

Particle size distribution. The particle sizing tests were made with University of Washington Mark III (Pilat) cascade impactors. The samples, taken at a point of average stack velocity and at isokinetic conditions, were collected at a sampling rate between 0.5 and 0.75 ft³/min. Stainless steel plates were used for the collection surface. When there was neither high temperature nor high acid content in the sample stream Apiezon grease was used to coat the collection plates in order to reduce bounce off or re-entrainment of particles.

Table II. Elemental composition of particulate emissions—zinc smelter.

	Vertical retort		Acid-sinter	
	Percent of total mass			
Element	Mean	Std. dev	Mean	Std. dev
Na	3.58	1.19	1.60	0.58
Mg	*		0.03	0.01
S	*		5.02	0.95
Cl	0.09	0.02	1.45	0.29
K	*		0.42	0.22
Ca	*		0.58	0.13
Fe	0.03	0.03	2.15	0.54
Cu	0.03	0.03	0.12	0.10
Zn	65.87	5.34	32.17	5.71
Cd	0.16	0.05	10.61	1.99
Sn	0.20	0.06	0.22	0.04
Sb	0.02	0.01	0.10	0.02
Pb	2.43	0.58	26.78	2.64

* Below detection limit.

Table I. Particulate emission rates and concentrations—zinc smelter.

Run no.	Facility sampled	Particulate concentration mg/Nm³ [a]	Volumetric flowrate Nm³/min	Emission rate kg/hr
5-4	Vertical retort	1101	700	46.3
5-5	Vertical retort	1177	684	48.3
5-2	Acid-sinter	138	1807	15.0
5-3	Acid-sinter	192	1751	20.3

[a] Nm³ = normal cubic meters at 760 mm Hg and 20°C, dry basis.

Analytical Methods

A Siemens MRS-3 multichannel wavelength dispersive X-ray fluorescence spectrometer with 15 fixed wavelength monochromators and one scanning channel to analyze for additional elements was used to determine the elemental composition of the characterization and impactor stage samples. The method, described previously,[6] requires that the aerosols be collected as light, uniform deposits on the substrate. Samples collected on the characterization filters were analyzed directly; samples collected on cascade impactor stages were redeposited as

Table III. Particle size distribution summary—zinc smelter.

Test no.	Facility	Mass median diameter micrometers	Percent less than 2.5 micrometers	Percent less than 10 micrometers
P6	Vertical retort	1.8	65	98+
P7	Vertical retort	2.2	56	98+
P4	Acid-sinter	2.1	52	76
P5	Acid-sinter	0.9	68	81

Table IV. Particulate emission rates and concentrations—lead smelter.

Run no.	Facility sampled	Particulate concentration mg/Nm³ [a]	Volumetric flowrate Nm³/min	Emission rate kg/hr
5-1	Blast furnace	6.70	11510	4.62
5-2	Blast furnace	7.32	12490	5.47
5-3	Blast furnace	6.28	12050	4.54
5-4	Blast furnace	3.67	11860	2.61
A-1	Acid-sinter	16.59	3950	3.93
A-2	Acid-sinter	71.03	3510	14.93
A-3	Acid-sinter	58.46	3780	11.84
A-4	Acid-sinter	4.48	3610	0.94
A-5	Acid-sinter	11.61	3700	2.58

[a] Nm^3 = normal cubic meters at 760 mm Hg and and 20°C, dry basis.

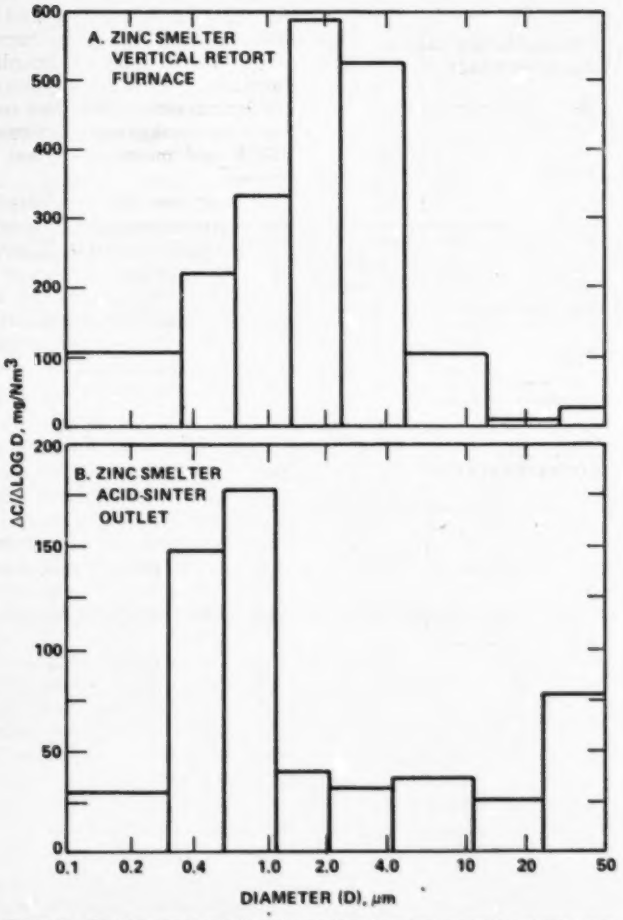

Figure 2. Particle size distribution of emissions from the vertical retort (A) and the acid sinter stacks of the zinc smelter.

thin layers since the impactors yield small piles on the stages that would result in internal absorption of the XRF characteristic fluorescence emissions. The material on each impactor stage was suspended in liquid and subsequently redeposited on polycarbonate films.[7]

Results

The results of the emissions tests including mass emission rates, elemental composition of the particulate emissions obtained by X-ray fluorescence analysis, and particle size distributions are presented below for each of the three smelter types.

Zinc Smelter

Mass emission rates. The particulate emission rates and concentrations obtained at the zinc smelter with EPA Method 5 are tabulated in Table I. Two tests were made at the vertical retort stack and two tests were conducted at the acid-sinter plant baghouse outlet. The average particulate concentration in the retort stack emissions was 1138 milligrams per dry standard cubic meter and in the acid plant outlet was 165 milligrams per dry standard cubic meter.

Chemical characterization. The elemental composition as determined by XRF analysis of collections on fluorocarbon type characterization filters are presented in Table II for the two emission points tested at the zinc smelter. Because light elements such as carbon and oxygen could not be determined by XRF, the total masses do not equal 100 percent. The results from the testing of the outlet side of the baghouse controlling emissions from the sintering and acid plant operations are based on tests taken over a two day period and are the average of 13 filter analyses. The elemental composition results of vertical retort emissions were obtained from 12 filter analyses.

The acid-sinter outlet emissions show high zinc content (over 32 percent) with the lead content almost as high (26 percent). The vertical retort emissions have a higher percentage of zinc (65 percent) which is much higher than that of the next most abundant base metal, lead (2.4 percent). The particulate emissions from the acid-sinter outlet had a sulfur content of about 5 percent.

Particle size distribution. The particle size measurements obtained with Pilat in-stack impactors are summarized in Table III. From cumulative plots of the mass median diameter (MMD), the percent less than 2.5 μm, and the percent less than 10 μm were

Table V. Elemental composition of particulate emissions—lead smelter.

	Blast furnace		Acid-sinter plant	
	Percent of total mass			
Element	Mean	Std. dev	Mean	Std. dev
Na	2.65	0.34	0.25	0.04
Mg	*			
S	3.47	0.48	5.28	0.61
Cl	1.19	0.47	3.24	0.68
K	0.23	0.23	0.56	0.09
Ca	*		*	
Fe	*		*	
Cu	*		0.10	0.06
Zn	15.30	2.08	0.22	0.11
Cd	3.02	1.09	5.28	1.05
Sn	*		*	
Sb	*		*	
Pb	58.06	12.04	49.75	6.87

* Below detection limit.

Table VI. Particle size distribution summary—lead smelter.

		Mass median diameter	Percent less than 2.5	Percent less than 10
Test no.	Facility	micrometers	micrometers	micrometers
P1	Blast furnace	1.2	77	90
P2	Blast furnace	1.2	75	91
P3	Blast furnace	1.2	73	85
P4	Blast furnace	0.9	78	88
P5	Acid plant	0.95	55	75
P8	Acid plant	0.88	67	77
P9	Acid plant	<.1	85	90

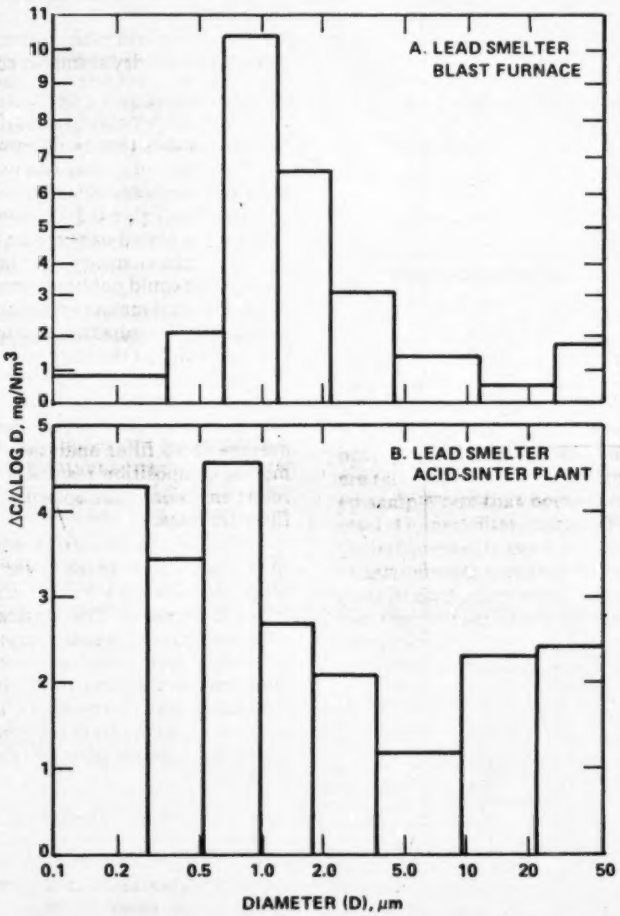

Figure 3. Particle size distribution of emissions from the blast furnace (A) and the acid sinter (B) stacks of the lead smelter.

determined. The average values obtained from two runs at each facility yielded a 2.0 μm MMD at the vertical retort with 60 percent less than 2.5 μm and 98 percent less than 10 μm. At the acid sinter stack, the average MMD was 1.5 μm with 60 percent less than 2.5 μm and 78 percent less than 10 μm. At the vertical retort stack, a series of five extractive tests with Andersen ambient impactors were also made to determine particle size. These tests yielded the following results which agree well with the in-stack impactor measurements: MMD 1.6 μm; less than 2.5, 67 percent; and less than 10 μm, 95 percent.

Particle size distribution of emissions from the zinc smelter are shown graphically for the in-stack samples collected at the retort and acid-sinter facilities in Figure 2.

Lead Smelter

Mass emission rates. The results of the Method 5 tests to determine particulate emission rates and concentrations at the lead smelter are shown in Table IV. Testing was conducted first at an outlet of the baghouse that controlled emissions from the blast furnace. Four Method 5 tests were run simultaneously with the characterization and the particle size sampling. The particulate concentration averaged 6.0 milligrams per dry standard cubic meter at an average stack temperature of 154°F and moisture content of 1.56 percent.

Testing was also conducted at the combined outlet duct of the acid plant and two baghouses which control emissions from the back section of the sinter plant. Five tests at this location yielded an average concentration of 32.5 milligrams per dry cubic meter with an average stack temperature of 168°F and a moisture content of 4.87 percent.

Chemical characterization. The elemental composition of the lead smelter emissions from the blast furnace and acid-sinter facilities are shown in Table V. As expected, lead is the predominant element present at both emission points. The average zinc content in the blast furnace emissions is about 15 percent compared to only 0.22 percent in the acid-sinter outlet. The average results for the blast furnace were obtained from the analysis of 13 fluorocarbon type filters taken during the same period as the four Method 5 tests, the acid-sinter results were obtained from 11 filter samples taken during the first three Method 5 tests.

Particle size distribution. The lead smelter particle size measurements are summarized in Table VI. While both the blast furnace and acid-sinter outlet had small mass median diameters of

Table VII. Particulate emission rates and concentrations—copper smelter.

Run no.	Facility sampled	Particulate concentration mg/Nm³ [a]	Volumetric flowrate Nm³/min	Emission rate kg/hr
P1	Reverberatory furnace	2486	3136	468
P2	Reverberatory furnace	2311	3169	439
P3	Reverberatory furnace[b]	563	2456	83
P4	Acid plant	30.7	1972	3.6
P5	Acid plant	74.6	1969	8.8

[a] Nm^3 = normal cubic meters at 760 mm Hg and 20°C, dry basis.
[b] Approximately 50–80 percent cutback on plant.

Table VIII. Elemental composition of particulate emissions—copper smelter.

	Reverberatory furnace		Acid-plant	
	Weight fraction ×10⁴			
Element	Mean	Std. dev	Mean	Std. dev
Na	0.14	0.08	*	
Mg	0.04	0.01		
Al	0.04	0.07	0.02	0.03
Si	0.19	0.26	*	
P	0.18	0.29	0.66	0.37
S	264.0	92.0	59.6	6.6
Cl	0.9	0.5	0.05	0.06
Ca	0.8	0.6	*	
Fe	10.6	5.3	*	
Cu	35.4	10.0	*	
Zn	5.4	1.7	*	
As	11.6	2.8	*	
Se	7.3	1.2	*	
Cd	0.02	0.02	*	
Sn	0.7	0.3	*	
Pb	8.3	2.9	*	

* Below detection limit.

Table IX. Particle size distribution summary—copper smelter.

Test no.	Facility samples	Mass median diameter micrometers	Percent less than 2.5 micrometers	Percent less than 10 micrometers
MC1	Reverberatory furnace	0.88	67	86
MC2	Reverberatory furnace	0.85	68	86
MC3[a]	Reverberatory furnace	<0.25	86	92
MC4	Acid plant	<0.10	92	95

[a] The MC3 sample was taken during reduced plant load.

about one micrometer, the distributions of the size were different as shown in Figure 3. The blast furnace particle distribution has a prominent peak of about 1 μm, whereas the acid-sinter emissions had more nearly the same mass on all of the impactor stages.

The impactor samples from the lead smelter were examined by redeposition of the particles in uniform layers and analysis of XRF. Of the four most abundant metals in the furnace emissions (lead, zinc, cadmium and iron), the first three were similar in size distribution to that of the total mass (Figure 3A) with about 50 percent of the total amount of each element on the sixth stage (D_{50} = 0.66 μm) alone. Iron was present on all stages and had a larger MMD of about 5 μm. The MMD of the emissions from the acid sinter outlet was less than 1 μm for all elements determined by XRF except iron, silicon, and aluminum.

Copper Smelter

Mass emission rates. The summary of the five Method 5 particulate emission tests run at the copper smelter is shown in Table VII. Three tests were conducted on emissions from the reverberatory furnace. During the first two tests on the furnace, the particulate concentration averaged 2400 milligrams per dry standard cubic meter with an average temperature of 522°F and a moisture content of 9.7 percent. The sulfur dioxide concentration averaged 5700 ppm. The last furnace test was under a system cutback with reduced emissions.

Only two particulate tests were made on the acid plant stack. The average particulate mass concentration was 53 milligrams per dry cubic meter. Stack temperature was 165°F and the average moisture content was 0 percent.

Chemical characterization. The XRF analyses of the fluorocarbon filters collected at the copper smelter are presented in Table VIII. The results are expressed as weight fraction rather than percentage because most of the collected mass was not analyzable by XRF, presumably due to a large presence of low atomic number elements (oxygen, carbon, etc.). Although many elements were detected in the reverberatory furnace emissions, the concentrations were very low. The most abundant metal, copper, had only an average concentration of 8.4 mg/Nm^3 in the 24 samples. Of the elements determined, only sulfur (0.6 percent) was significant in the acid plant emissions.

Particle size distribution. Three particle size measurements with in-stack impactors were made at the copper reverberatory furnace stack and one measurement at the acid plant. The results are summarized in Table IX.

The most abundant elements determined by XRF on all impactor stages in the reverberatory furnace emissions were iron, copper and sulfur. The elements copper, zinc, lead, and sulfur were predominantly submicron in size, other elements such as iron, silicon, calcium and aluminum were distributed more evenly throughout the impactor stages with mass median diameter sizes greater than one micron. At the copper smelter acid plant, where only one sample was taken due to a shutdown, the particle size was extremely small with over 70 percent of the mass located on the final impactor stage (Figure 4B).

Conclusions

The sampling and analytical methods employed previously to physically and chemically characterize emissions from other stationary sources have been successfully applied to three nonferrous smelters—a zinc, lead, and copper ore processing plant. Although the particulate emissions from the baghouses were relatively high during the testing, the bags appeared to be in good condition. Reverberatory furnaces at almost all copper smelters have been replaced, so the results obtained at the sampled copper smelter is not necessarily representative of most copper

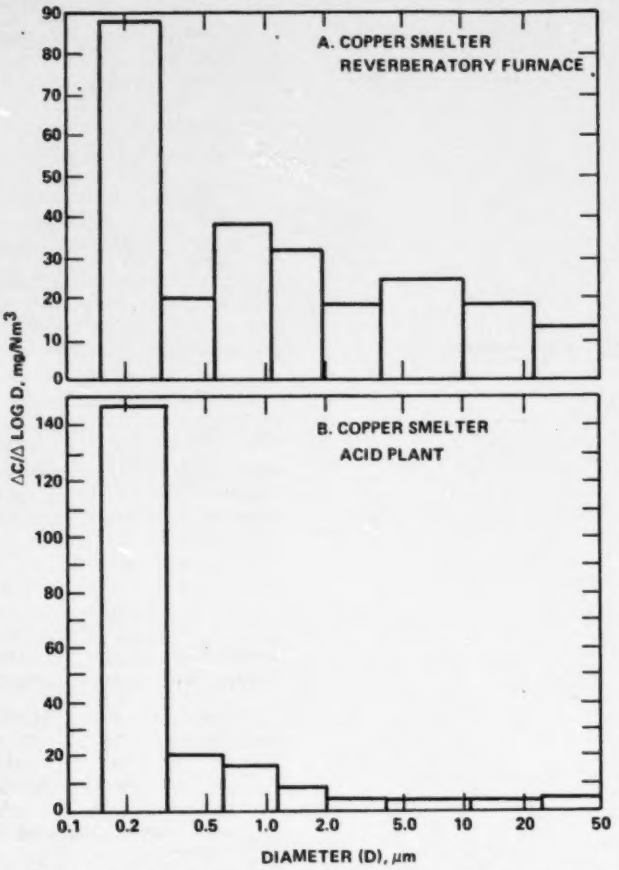

Figure 4. Particle size distribution of emissions from the reverberatory furnace (A) and the acid plant (B) of the copper smelter.

smelters. The elemental composition measurements should be useful in source apportionment and other air pollution research projects. The overall results of the particle size measurements have shown that mass median diameters of the particulate emissions are small (near one micrometer or less) at both the furnace and the acid plant stacks at all three of these smelters.

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